



Date: January 26, 1982

Subject: Primary Processes R & D - Tucson  
Monthly Report - January

From/Location: E. L. Cambridge

To/Location: J. G. Kaufman

#### BASIC RESOURCES RESEARCH

##### AD-117 Impurity Leaching Process

Experimental work on the removal of impurities from alumina produced by the HCl/Clay process is progressing. A two-level, fractional factorial design of experiments assessing the effect of nine independent variables on the leaching of impurities has been carried out. Samples are now out for analysis and evaluation of the dependent variables.

Calorimetric methods of analysis for  $P_2O_5$  in alumina and clay are being evaluated for use by the analytical laboratory for the near term.

##### AD-105 Alumina Process

Preparation of samples of clay for use in the AD-105 project is complete. A set of eight experiments designed to test factors important in the clay leach and hydrolysis steps has been begun. A literature search for material relevant to the project has been started.

##### AD-XXX $AlCl_3$ Process

A first campaign of 16 statistically designed experiments is being carried out to determine the importance of five independent variables; temperature, calcination time, ACH particle size, heating rate and carrier gas on the thermal decomposition of aluminum chloride hexahydrate (ACH). The key dependent variables that will be measured are residual hydrogen, residual chloride and surface area.

Partial calcination of green petroleum coke to produce an active reductant for the reductive chlorination of partially calcined ACH was investigated. Eight fractional factorial design experiments were completed with temperature, calcination time, and particle size as the independent variables. Residual hydrogen and surface area are the dependent variables. Samples have been sent to Columbia Falls for surface area determination. The carbon value of the green petroleum coke was about 83% for all eight experiments.

Partial calcination of green petroleum coke together with ACH and green petroleum coke under controlled oxygen atmosphere were also carried out. Samples have been submitted for residual hydrogen and surface area measurements.

Chlorination experiments of partially calcined ACH (calcined at 400°C) were conducted in a fluidized bed reactor; 1½" diameter with a bed height of about 3". Preliminary results indicated that about double the chlorination rate was obtained using partially calcined ACH with partially calcined petroleum coke (calcined at 650°C, for 30 minutes) as compared to using cell-grade alumina with partially calcined petroleum coke. The highest chlorination rate was observed using the partially calcined petroleum coke calcined under controlled oxygen atmosphere. A gas chromatograph is being set up on-line with the fluidized bed reactor to quantitatively determine the kinetics of the reductive chlorination of the partially calcined ACH.

Pretreating of both fully calcined coke and partially calcined coke with acidified  $AlCl_3$  melts is being conducted to determine if rate of chlorination can be enhanced and if CO can be made by reaction of  $CO_2$  with the treated coke.

#### REDUCTION RESEARCH

##### AD-108 Process

Anodes have been successfully made in a prebaked form using trihydrate dried at 110°C, trihydrate partially calcined to 400°C, and MGA, and then baking. The 110°C and 400°C trihydrate anodes were molded with vent holes. One set of 400°C anodes did not have vent holes. All anodes were of excellent quality after baking, indicating that precalcining of trihydrate is not required for producing sound anodes provided vent holes are incorporated in 110°C dried material. The vent holes can serve as aluminum conductor paths.

Trihydrate has been calcined to 700°C but not yet molded into anodes. All anodes will then be characterized with respect to residual hydrogen, density and performance in electrolysis.

Carbon coated  $Al_2O_3$  powder has been prepared in the desired C/ $Al_2O_3$  ratio and tested in electrolysis by floating on the surface of the electrolyte. Good electrical contact to the powder anode could not be achieved. Porous graphite has been received and will be used as a diaphragm and electrical contact to the powder anode.

Additional diaphragm materials of BN and AlN prepared from sintered powder and aluminum foam converted to AlN have been ordered for further evaluating the powdered anode-diaphragm concept.

##### AD-119 Modified Electrolytes

A GenRad Model 1688 digibridge was selected for conductivity measurement of molten salt electrolyte for composite anode work (AD-108) and for electrolyte modification studies using LiF. The unit was tested using an aqueous electrolyte with resistances comparable to those encountered in molten salts work ( $\approx 0.5\Omega$ ). A frequency greater than about 4 kHz was found to be required to eliminate the electrode capacitive effect on resistant measurements. A conductivity probe, made of BN with  $TiB_2$  electrodes, was designed and is being

fabricated for use in conjunction with the digibridge for molten salt conductivity studies.

#### AD-114 Al<sub>2</sub>S<sub>3</sub> Processes

A computer code, procured from Argonne National Laboratory, was loaded on Denver computer and it is now available for use. This code calculates the thermodynamic equilibrium composition using free energies minimization techniques. This code is a valuable tool in evaluating alternative processes, and for identifying "a priori" the important operating variables. The code will initially be used to evaluate various alternative routes for the synthesis of aluminum sulfide.

#### AD-116 Potlining Resource Recovery

The first meeting of the Alcan-Anaconda joint evaluation group met in Tucson, January 12-13. Criteria for process selection and evaluation were developed and applied to 30<sup>+</sup> processes. The Mimi-L and D processes from Alcan and the Anaconda AD-116 process were selected for further consideration. Detailed capital and operating costs will be developed and reviewed at the next meeting in Kingston in late February.

#### Petroleum Pitch

Results from tests of Ashland-240 pitch in Soderberg composites at Columbia Falls indicate poor baked composite quality which is attributed to a Conradson coking value of 48% in contrast to the Ashland specification of 52%. Ashland has been contacted and some of the pitch is being returned to Ashland for examination.

Arco pitch from the last Lummus (New Jersey) pilot plant run has just arrived at Harvey Technical Center. This material is supposed to be nearest to commercial quality pitch of all samples produced to date, and was the last pitch produced before Arco cancelled the needle coke project. A 30-lb sample of this pitch will be sent to Columbia Falls next week for pitch property value determination.

#### Alternative Cokes

Initial contacts have been made with Arco Coal concerning use of purified coal as a potential source of aluminum industry carbon. Some preliminary coal property data have been received and more requested.

#### DEVELOPMENT & TECHNICAL SERVICES

##### Coke & Pitch Quality

A formal report quantifying the effects of vanadium and sulfur contents of calcined petroleum coke and QI content of coal-tar pitch on operating costs for prebake Hall-Heroult cells is now being printed for issue next week.

#### Lithium Fluoride

The experimental cells at Sebree experienced improved current efficiency in December (91.66% - up from 88%) as a result of better process control. Problems in determining the chemical analysis of lithium electrolyte have been solved.

Tucson has requested Louisville to conduct a literature search on the use of lithium fluoride in aluminum reduction cells. Results are expected within a week. The literature search, combined with an economic analysis on  $\text{AlF}_3$ , will be the basis of a position paper to be submitted by mid-March.

#### Sebree Large Anode Project

The project is currently in the cell stabilization and voltage reduction phase. Large anode cells have shown improved stability as compared to the control cells. No major problems in pot operation have occurred. The last large anodes will be set on February 10. Test data will continue through March.

#### Fort Meade $\text{AlF}_3$

Ninety tons of Fort Meade aluminum fluoride are now in storage tanks at Sebree. This is enough material to conduct a one-month, one-potline test which is scheduled to begin February 1.

#### Columbia Falls Bus Modifications

The recommendation for bus modification at Columbia Falls was received from ASV and is now under study at Tucson and Columbia Falls.

#### Arco Fines

Five hundred tons of Cherry Point fines have been used at Sebree as substitute ball-mill product. No difference in anode quality was measured. Problems were experienced with dust collectors plugging.

#### Tucson Computer Requirements

A study to define Tucson's initial computer requirements has begun in conjunction with Joe Palmer's overall efforts and will be completed within two months.

#### Skirt/Flange Corrosion - Columbia Falls

Initial coating compositions were selected for skirts and flanges and discussed with METCO technical representatives. The field service engineer for METCO was then contacted and put in touch with Dave Krouse to work out details for scheduling coating application for evaluation at Columbia Falls. The initial coatings selected for skirts and flanges were a pure aluminum with low (a few percent)  $\text{Al}_2\text{O}_3$  and a higher percent  $\text{Al}_2\text{O}_3$  (approximately 10%). Alloying of coating compositions, which will undoubtedly improve corrosion

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resistance as well as cost, will not be initiated until the Al-Al<sub>2</sub>O<sub>3</sub> composition results are deemed unsatisfactory.

PERSONNEL

Katherine J. Gray was hired as a Technician in the Basic Resources group, effective January 25, 1982.

OTHER

Following approval of the Carbon & Reduction Research Lab AFE, the construction contract was awarded to Pace Construction Inc. of Tucson. Site work began on January 15 with building completion scheduled for mid-May. Equipment procurement procedures have been initiated.

A meeting was held with Minerals company personnel to further define our specific analytical requirements. Arrangements are in progress to obtain the necessary methods, standards and equipment.

  
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